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Enrichment of polyphenolic compounds from grape leaf extract (*Vitis vinifera* L.) using macroporous resin and investigating its adsorption kinetics and dynamics

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ABSTRACT

Vitis vinifera L., with the common name "grape", is native to Mediterranean region and widely grown in different climatic conditions all over the world. Grape leaves are also a rich source of important polyphenolic compounds that have been recognized for their diverse promising health benefits and therapeutic properties. In this study, the adsorption/desorption process on LXA-10 resin was investigated to enrich polyphenolic compounds in the prepared hydroalcoholic extract of grape leaves. The extract was obtained using ultrasound-assisted probe with water and ethanol (50:50) with an extraction yield of 26.4%. The pH value of the extract for maximum adsorption was optimized at 3.26. According to the findings of this study, the kinetic adsorption fitted well with pseudo-second-order model (R^2 = 0.99). Moreover, the dynamic study revealed the adsorption capacity and required solvent for polyphenolic desorption being 42 mL and six-bed volume (BV), respectively. In the enriched powder, the amount of quercetin-3-*O*-glucuronide and quercetin-3-*O*-glucoside increased from the ranges of 19.43 to 45.92 mg.g^{-1} and 41.244 to 72.88 mg.g^{-1}, respectively. The antioxidant activity of enriched polyphenols also increased from 252.37 in the initial grape extract to 408.03 mM iron (II).mg^{-1}.

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1. Introduction

Vitis vinifera L., a valuable plant belonging to the Vitaceae family, is considered as one of the important fruit crops in the world from commercial point of view. The wastage problem regarding grape processing by-products has attracted high public attention, as well. A literature survey demonstrates that a large number of reports have mainly focused on the processing of valuable products from different organs of grapes, *e.g.*, seeds, stems, and skin. However, only a few studies have discussed the case from the leaves and tendrils of *V. vinifera* so far (Moldovan et al., 2020). Grape leaves are recognized as one of the potential nutritional sources due to having phenolic acids, and organic acids, *e.g.*, malic, oxalic, fumaric, ascorbic, citric, and tartaric acid, tannins, flavonols, anthocyanins, vitamins, and carotenoids. Most of the therapeutic properties of the plant may be attributed to its polyphenolic compounds. Regarding the various pharmacological effects, *e.g.*, antimicrobial, anti-inflammatory, and antioxidant activities of a wide variety of phenolic compounds, they have received considerable attention (Ergun et al., 2009).

Macroporous adsorption resins are polymeric adsorption ones having polar, slightly polar, or non-polar, characteristics and capable of separating and enriching active compounds from a broad spectrum of natural sources. It has been well documented that surface areas and pore sizes are among the main physical properties of the resins affecting their interaction with the target molecules (Seif Zadeh and Zeppa, 2022). Generally, the active molecule could be adsorbed on the surface of the used resin based on two types of mechanisms, namely physical and chemical adsorption mechanisms. In the physical adsorption, weak forces like Van der Waals

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and electrostatic attraction occur between the active molecules and the employed adsorbent, while in the chemical interactions, the molecules are adsorbed on the surface of the resin through strong covalent bonds (Rathi and Kumar, 2021).

There are several macroporous resins to enrich polyphenolic compounds from natural sources, such as XAD-16 resin for recovery and concentration of polyphenols from roasted hazelnut skin extract (Seif Zadeh and Zeppa, 2022), XAD-7HP for enriching blueberry poly phenols (Meng et al., 2017), and X-5 macroporous resin to purify *Sphallerocarpus gracilis* stem and leaves polyphenols (Ma et al., 2015). However, to the best of our knowledge, no reports could be found in the literature for the enrichment of polyphenolic compounds of grape red leaves.

In this project, the grape leaves were first air-dried and subsequently ground to a fine powder. In the next step, the polyphenolic compounds were extracted from the grape leaf powder using a mixture of water and ethanol as the solvent using ultrasound-assisted prob. In the other part of this study, the performance of three different types of the macroporous resin has been investigated for the enrichment of the grape leaf extract. The effective adsorption parameters like extract pH value, kinetic and dynamic have also been studied, and the enriched polyphenolic compounds of grape leaves extract have been subjected to the HPLC-PDA analysis. Finally, the antioxidant activity of the crude extract and polyphenolic enriched compounds has been evaluated.

2. Experimental

2.1. Reagents and materials

Sodium carbonate (Na_2CO_3), Folin-Ciocalteu, ascorbic acid ($C_6H_8O_6$), sodium hydroxide (NaOH), 2,4,6-tripyridyl-s-triazine (TPTZ), iron(III) chloride hexahydrate ($FeCl_3.6H_2O$), iron(II) sulfate heptahydrate ($FeSO_4.7H_2O$), aluminum chloride ($AlCl_3$), sodium nitrite ($NaNO_2$), dimethyl sulfoxide (DMSO) and HPLC grade methanol (MeOH) were all purchased from Merck (Darmstadt, Hesse, Germany). Ethanol (EtOH) for HPLC analysis, and hydrochloric acid (HCl) were obtained from Fisher Chemical (Waltham, Massachusetts, U.S). The LXA-10, AB-8, and HP-20 macroporous adsorbent resins were purchased from Sunresin (Xi'an, Shaanxi, China) and their physical properties are presented in Table 1. The HPLC grade water was supplied by a Direct-Q3 UV Millipore water purification system (Millipore, Molsheim, France). The quercetin-3-*O*-glucuronide, quercetin-3-*O*-glucoside, and gallic acid (GA) were respectively obtained from Phytolab (Germany) and Phytopurify (Chengdu, Sichuan, China).

Table 1
Physical properties of macroporous resins.

Type of resin	Surface area ($m^2.g^{-1}$)	Pore size (nm)	Polarity
HP-20	590	52	Weak
LXA-10	> 400	8.14	Medium
AB-8	480-520	130-140	Weak-polar

2.2. Extraction process

The dried grape leaves were collected from Sardasht in the southwest of West Azerbaijan Province and transferred to the Medicinal Plants and Drug Research Institute, Shahid Beheshti University, Tehran for more analysis. The polyphenolic compounds of the grape leaves were extracted using ultrasonic-assisted probe method as described previously (Tacchini et al., 2019). Accordingly, the dried powdered of grape leaves (25 g) were extracted with 500 mL of aqueous ethanol (50:50) with power 50% for 10 minutes at a constant temperature (30 °C). After extraction, the obtained extract was filtered using a centrifuge and completely dried with a rotary evaporator. The obtained extract stored at 4 °C for more analysis.

2.3. Total phenolic content (TPC) determination

The TPC of the extract was determined based on the Folin-Ciocalteu reagent (FCR) (Bayati et al., 2021). Briefly, 125 µL of diluted FCR (1:9 in water) and 100 µL of sodium carbonate (7.5% w/w) solution were added to 25 µL of diluted sample (1000 ppm). After adding reagent, the plate was stored for 2 h and the absorbance was measured at 760 nm by a multifunctional microplate reader. The results were expressed in mg gallic acid (GA) equivalent per gram of sample dry weight.

2.4. Total flavonoids content determination

Total flavonoids was determined following a previously reported method (Mohammadi et al., 2022). Based on this method, in the first step, 25 µL of diluted sample, 100 µL of water and 7 µL of sodium nitrite (5%) were added into a 96-multiwell plate. After 6 min, 7.5 µL of aluminum chloride (10%), 100 µL of sodium hydroxide (4%), and 10 µL of deionized water were added to the plate and incubated for 15 min. The absorbance of solution was finally measured at 510 nm using a multifunctional microplate reader. The results were expressed in milligram based equivalent per gram of rutin on sample dry weight.

2.5. Total carbohydrate content determination

The total carbohydrate content was determined using phenol-sulfuric acid method (Nielsen, 2010). Briefly, 1 mL aliquot of the sample was mixed with 5 mL sulfuric acid (96%) and 1 mL phenol (5%) and the mixture was allowed to stand for 20 min at 30 °C. After 20 min, the absorbance of the mixture was recorded at 490 nm. The blank solutions were also prepared in identical manner as above, except that the 2 mL aliquot of sample was replaced by distilled water. Finally, the total carbohydrate content was expressed based on the µg glucose equivalent per mL sample.

2.6. Antioxidant activity

The antioxidant activity of the extract was measured using the ferric reducing antioxidant power (FRAP) assay (Alam et al., 2021). Briefly, to prepare FRAP reagent, 25

mL of acetate buffer (300 mM solution, pH 3.6) was mixed with 2.5 mL 2,4,6-tri(2-pyridyl)-1,3,5-triazine or TPTZ (10 mM solution) and 2.5 mL of $FeCl_3.6H_2O$ (20 mM solution) in 50 mL HCl solution (60 mM solution). To evaluate the antioxidant activity, 200 µL of the fresh FRAP reagent was added to 20 µL of each sample and the resulting mixture was incubated at 37 °C for 30 min. Immediately after, the absorbance was measured at 593 nm using a microplate reader, EPOCH2 Bio Tek (VT, USA). The results were reported based on the iron(II) sulfate calibration curve and expressed as mM iron(II) sulfate equivalents.mg^{-1}. Ascorbic acid as a natural antioxidant in food sources was considered as a positive control.

2.7. High-performance liquid chromatography (HPLC) analysis

To quantify the amount of quercetin-3-*O*-glucuronide and quercetin-3-*O*-glucoside, as two main flavonoids of the grape leaves extract, HPLC analysis was performed using a Waters Alliance 2695 HPLC system (Milford, Massachusetts, USA) coupled with a photodiode array (PDA) detector 996, and equipped with SunFire C18 column (150 mm × 5.0 mm × 3.5 µm) at 30 °C. Linear gradient elution system was used with acetonitrile (A) and water (B) as eluents as follows: 0-20 min, 10-70% A; 20-25 min, 70% A, 25-30 min, 70-100% A; 30-34 min, 100% A, 34-35 min, 100-10% A, 35-40 min, 10% A. The flow rate of the mobile phase (eluent) was fixed at 1 $mL.min^{-1}$ and 20 µL of sample were injected into the system in each run and the chromatograms were monitored at 355 nm.

2.8. Macroporous resin pretreatment

To remove some monomers which can be trapped inside the pores of the synthesized resins, resin pretreatment step should be performed. For this purpose, at first, the resin was soaked in NaOH (4%) for 2-4 h. Then, the NaOH was removed and the resin completely washed with water. In the next step, the resin was soaked in 5% HCl for 2-4 h. After this process, the HCl was removed and resin washed with water. Finally, the resin was soaked in methanol for 2-4 h and then was washed with water until the pH value of the eluate reached 7.0. It should be noted that the water should be removed from resin before each usage.

2.9. Selecting the appropriate macroporous resin

To select the appropriate resin for enriching the polyphenolic compounds with the highest yield, three different resins, namely LXA-10, AB-8, and HP-20 were taken into consideration. For this purpose, the certain amount of each resin (20 g) was interacted with 30 mL of the obtained extract for 1 h. After reaching equilibrium, the adsorbent was soaked in water and was subsequently washed with absolute methanol. The amount of TPCs in the initial extract and the desorbed sample was determined using FCR reagent as described in section 2.3 and the appropriate resin was selected based on its potential to enrich polyphenolic compounds.

2.10. The pH value optimization

The batch adsorption study was performed to determine the optimum pH value of the extract for reaching the highest polyphenolic adsorption on the resin. For this purpose, 20 g resin was stirred with 30 mL of extract at three different pHs (2.28, 3.26: as the initial pH of the grape leaves extract and 4.27) for 1 h. After reaching equilibrium, the adsorption capacity (Chang et al., 2021) and adsorption percentage (Wieszczycka et al., 2020) were calculated based on the following equations:

$$\text{Adsorption } (\%) = \frac{C_0 - C_e}{C_0} \times 100 \qquad \text{(Eqn. 1)}$$

$$Q_e = (C_0 - C_e) \times \frac{V_i}{w} \qquad \text{(Eqn. 2)}$$

In the above equations, C_0 and C_e show the TPCs at initial and equilibrium ($mg.L^{-1}$), respectively, Q_e is adsorption capacity ($mg.g^{-1}$), the V_i represents the volume of the grape leaves extract (L), and w accounts for the mass of the resin (g).

2.11. Adsorption kinetic study

The kinetic study of phenolic adsorption on the selected resin was performed with stirring 30 mL grape leaves extract (pH 3.26) with 20 g of adsorbent at 100 rpm for 1 h. Aliquot samples were taken at different time intervals until reaching the equilibrium, and the TPCs were determined as described in the section 2.3. The adsorption kinetics was investigated using pseudo-first-order (Eqn. 3) and pseudo-second-order (Eqn. 4) models as follows (Capello et al., 2019):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad \text{(Eqn. 3)}$$

$$\frac{t}{q_t} = \frac{t}{q_e} + \frac{1}{k_2 q_e^2} \qquad \text{(Eqn. 4)}$$

The q_e ($mg.g^{-1}$) and q_t ($mg.g^{-1}$) are the amount of adsorbed phenolic compounds at equilibrium and in time t: k_1 (min^{-1}) and k_2 ($g.mg^{-1}.min^{-1}$) show the constant rate of pseudo-first-order and pseudo-second-order models, respectively.

2.12. Dynamic adsorption and desorption process

For performing dynamic analysis, a glass column (1.5 cm × 80 cm) was filled with 20 g of the selected resin. The grape leaves extract was loaded on the adsorbent and the TPC concentration of the outlet sample was investigated at different volume interval fractions (3 mL) for 1 h. The variation of C/C_0 against time was depicted as breakthrough curve which is required for the fixed-bed column scaling up. Breakthrough point time (t_{BP}) and exhaustion point time (t_{EP}), where the bed is saturated were calculated. In this study, the loading of the grape leaves extract was ended after reaching polyphenolic compounds percentage to the constant value in outlet sample (Heravi et al., 2022).
After the completion of the adsorption step and for the

desorption process, the adsorbent was first soaked with deionized water to eliminate some impurities and after that, the absolute methanol was loaded on the resin until no polyphenolic compounds were detected in the outlet sample.

3. Results and Discussion

3.1. Selection of a proper resin

To select the best adsorbent type for enriching polyphenolic compounds of the grape leaves extract, the ability of each resin was investigated based on the amount of the TPCs in the desorbed sample (Table 2). As can be seen, the LXA-10 resin with the highest amount of TPC (386 mg GA.g^{-1}) in desorbed sample showed a 2.4-fold increase in the enriching of polyphenolic compounds compared with the AB-8 and HP-20 resins with 1.29 and 2.02 fold increase, respectively. The adsorption capability of resins is generally related to the type of the target molecule and the nature of the adsorbent on which each target molecule could be adsorbed through different chemical, physical, and electrostatic interactions. Particularly, in this case, polyphenol compounds due to their various hydroxyl and benzene groups can be adsorbed on LXA-10 resin, which is more polar than two other types of the resin, through physical mechanisms like hydrogen bonds, and π-π conjugation interactions.

3.2. The pH value optimization

Fig. 1 shows the adsorption capacity and adsorption percentage of polyphenols at different pH values on LXA-10 resin. As seen, at pH = 3.26, which is the pH of the obtained extract, the highest amount of polyphenols was adsorbed onto the resin with adsorption percentage 66.78%. Moreover, the adsorption capacity reached its highest values, 0.107 mg.g^{-1} at pH = 3.26. In addition, anthocyanins are one of the phenolic compounds of grape extract which are stable at lower pH value (pH < 4) since they can form flavylium cation structures. However, as pH increases, their structure changes to the carbinol pseudo-base and quinoidal base which indicates the destruction of anthocyanin structures. This fact may be one of the reasons for the decrease in adsorption capacity and the relevant adsorption percentage (Pismenskaya et al., 2020).

3.3. Kinetic study of polyphenols adsorption on LXA-10 resin

The kinetic curves of adsorption polyphenolic compounds of the grape leaves extract on LXA-10 resin were depicted in Fig. 2. According to Fig. 2A, the adsorption capacity of polyphenols increased rapidly in the first 5 min. After that, the increase slowly continued up to 14 min, and finally at about 20 min reached equilibrium, with an adsorption capacity of 0.103 mg.g^{-1}. Table 3 shows the calculated parameters of the polyphenol's adsorption kinetics model on LXA-10 resin. In kinetic studies like isotherm models, the R^2 value and the conformity rate of the experimental adsorption capacity with its calculated value are essential parameters to determine the appropriate kinetic model. Based on the results, the pseudo-second-order model with the highest R^2 value (0.99) and its calculated adsorption capacity conformity (0.105 mg.g^{-1}) with the experimental data (0.103 mg.g^{-1}) was selected as the good kinetic model for describing the adsorption mechanism (Table 3). In conclusion, the adsorption process of polyphenolic compounds of grape leaves extract on the LXA-10 resin can be represented through pseudo-second-order model.

3.4. Dynamic adsorption and desorption tests

A breakthrough curve study can give an effective data about the behavior of polyphenols adsorption on the resin through fixed bed conditions (Fig. 3A). This data could be helpful for scaling up the adsorption column. The important parameters were estimated as follows. The breakpoint time (t_{BP}) of 4 min which shows the point when the ratio of polyphenols concentration in eluent reaches one-tenth of the initial loading extract, the ideal breakthrough curve (t_{IBC}) which shows the required time for full bed exhaustion is 14 min. After that, the concentration of polyphenolic compounds rises to exhaustion point time (t_{EP}) of 55 min.
For desorption polyphenolic compounds from the adsorbent, the resin was first soaked with 2-4 BV of water to eliminate some impurities like sugar, etc. Then, 10 BV of absolute methanol was utilized to separate phenolic compounds from the adsorbent resin. Based on the Fig. 3B, the TPCs compounds in outlet methanol increase significantly until 102 mL (5 BV) methanol, after that the amount of desorbed phenolic compounds is decreasing when using approximately 150 mL of methanol accounting for no significant polyphenolic compounds in the fractions representing the completion of the desorption process. The minimum solvent required to achieve the maximum adsorption of polyphenols on the surface of the resin can be considered as 120 mL (6 BV) (Fig. 3B). These results could be helpful for developing large-scale adsorption-desorption systems.

3.5. HPLC analysis of the enriched polyphenolic compounds of grape leaves extract

To investigate the ability of the resin to enrich polyphenolic compounds and calculate the enrichment factor more closely, the grape leaves extract before and after treatment with resin and enriched polyphenolic compounds of extract were quantitatively determined using HPLC-UV analysis based on the main components of red grape leaves like quercetin-3-*O*-glucuronide and quercetin-3-*O*-glucoside (Fig. 4). A simple perusal of the obtained chromatograms displays that after treating the initial grape leaves extract with the resin, the peaks related to polyphenolic compounds have been completely removed representing the high potential of LXA-10 resin to adsorb polyphenolic compounds of grape leaves extract (Fig. 5). Moreover, the amount of quercetin-3-*O*-glucuronide increased from 19.43 to 45.92 mg.g^{-1} and quercetin-3-*O*-glucoside increased from 41.24 to 72.88 mg.g^{-1} in enriched polyphenolic

Table 2
The comparison the amount of TPC (mg GA.g-1) in desorbed sample after treatment with different types of the resins.

Resin	Amount of TPC (mg GA.g-1)		
	Leaves extract	Desorbed sample	Desorbed sample/ leaves extract
LXA-10	135	386	2.85
AB-8	135	175	1.29
HP-20	135	273	2.02

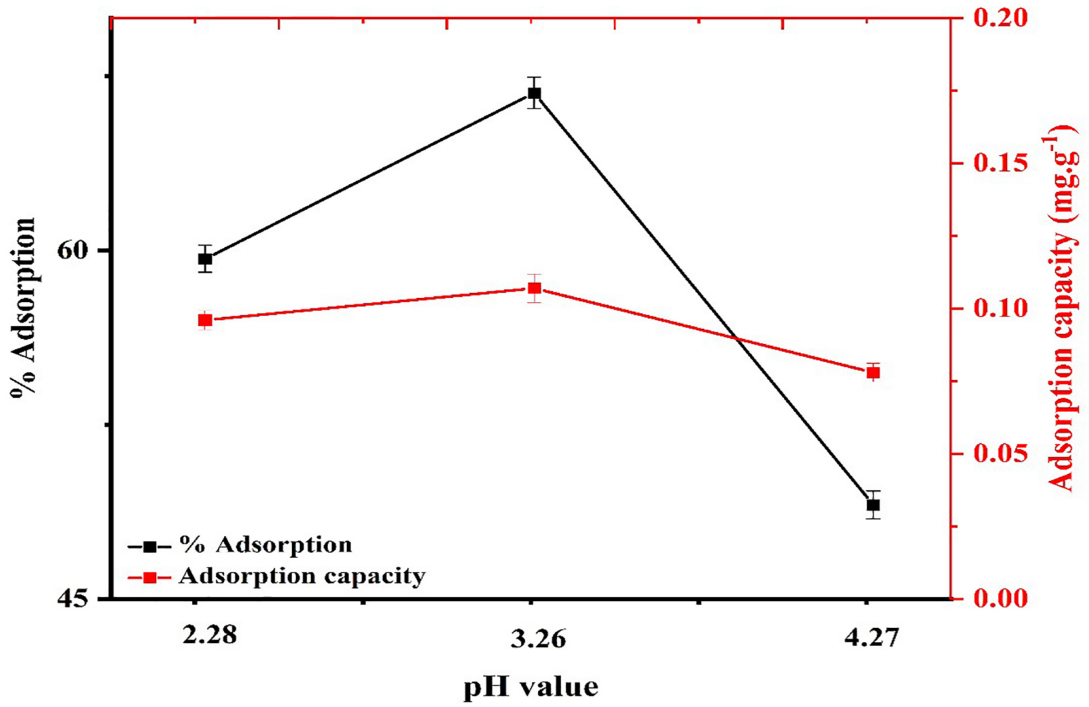

Fig. 1. Effect of pH value of the grape leaves extract on polyphenolic compounds adsorption characteristics on LXA-10 resin.

Table 3
Kinetic parameters for adsorption of polyphenolic compounds on LXA-10 resin.

Pseudo-first order model			Pseudo-second order model		
qe ($mg.g^{-1}$)	k_1(min^{-1})	R^2	qe ($mg.g^{-1}$)	k_2 ($g.mg^{-1}.min^{-1}$)	R^2
0.05	0.21	0.9	0.105	11.9	0.99

compounds sample in comparison with the initial grape leaves extract, respectively. Moreover, based on the phenol-sulfuric acid method, the total carbohydrate amount in the obtained polyphenolic enriched powder significantly decreased from 149.42 ± 0.04 to 61.96 ± 0.02 µg glucose.mL^{-1} sample which shows the good sugary impurities elimination from initial grape leaves extract.

3.6. Antioxidant activity

Antioxidant activity of the enriched polyphenolic compounds of grape leaves extract and its comparison with extract after treating with resin was investigated using FRAP test. The FRAP test is based on the reducing the ferric-TPTZ (Fe(III)-TPTZ) complex to the blue ferrous-TPTZ (Fe(II)-TPTZ) complex in the presence of

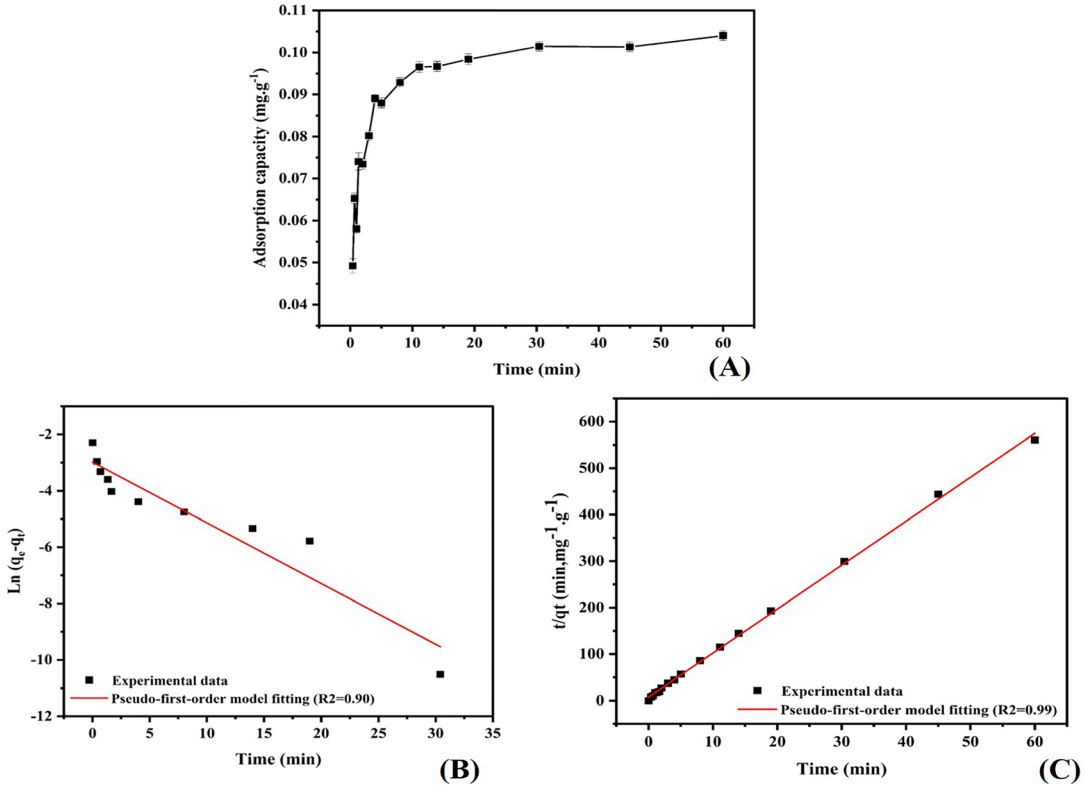

Fig. 2. (A) The kinetic curve and (B) Pseudo-first-order, (C) Pseudo-second order model plots for polyphenolic compounds adsorption from grape leaves on LXA-10 resin.

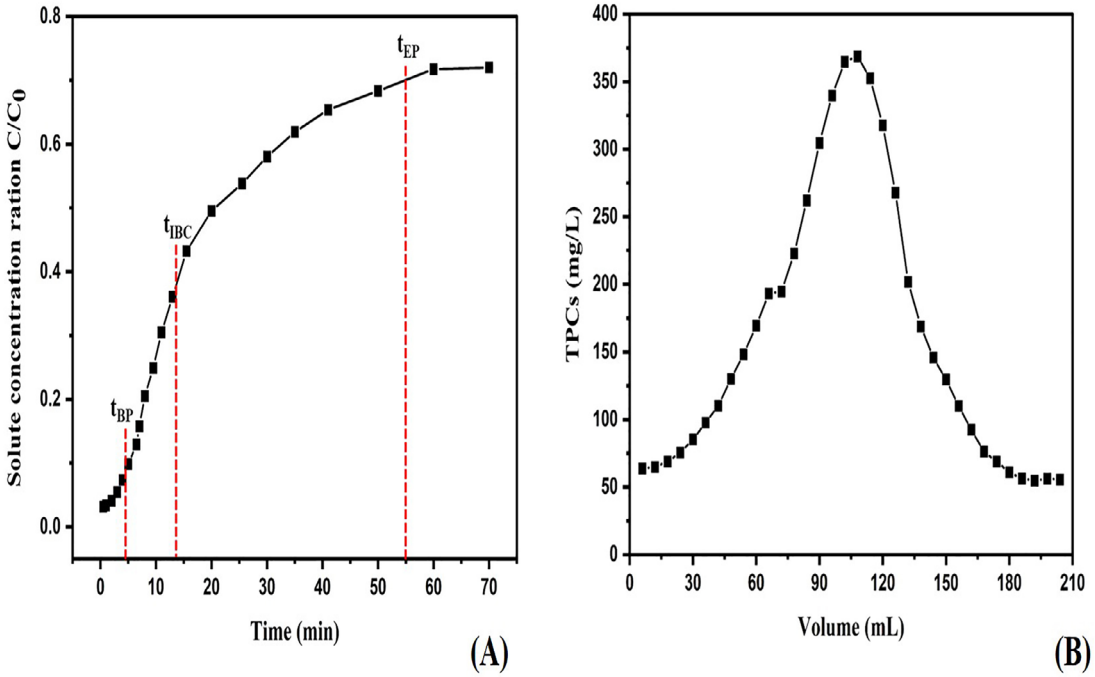

Fig. 3. The (**A**) breakthrough curve and (**B**) desorption curve of polyphenolic compounds on LXA-10 resin.

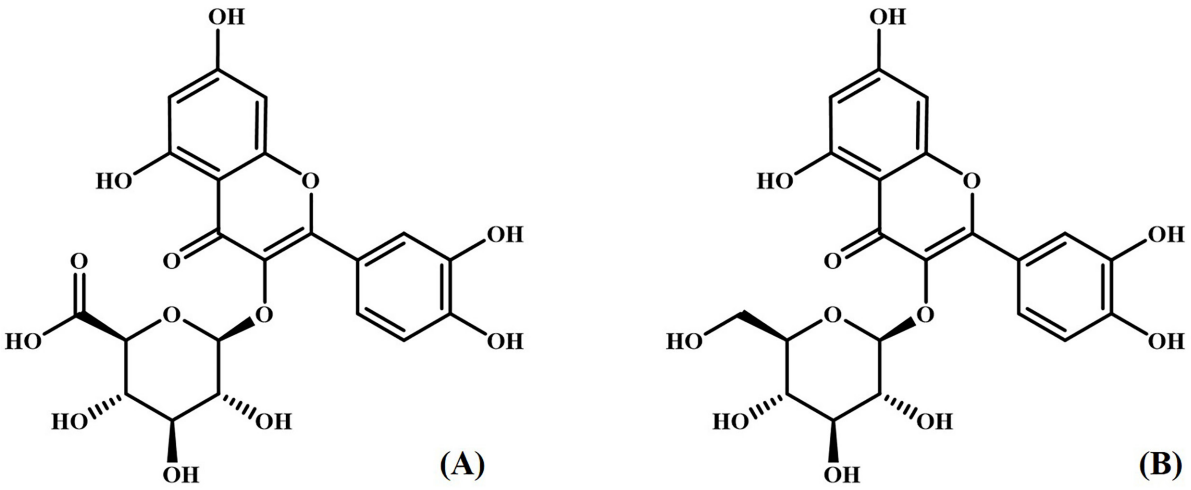

Fig. 4. The structure of (A) quercetin-3-*O*-glucuronide and (B) quercetin-3-*O*-glucoside in grape leaves extract.

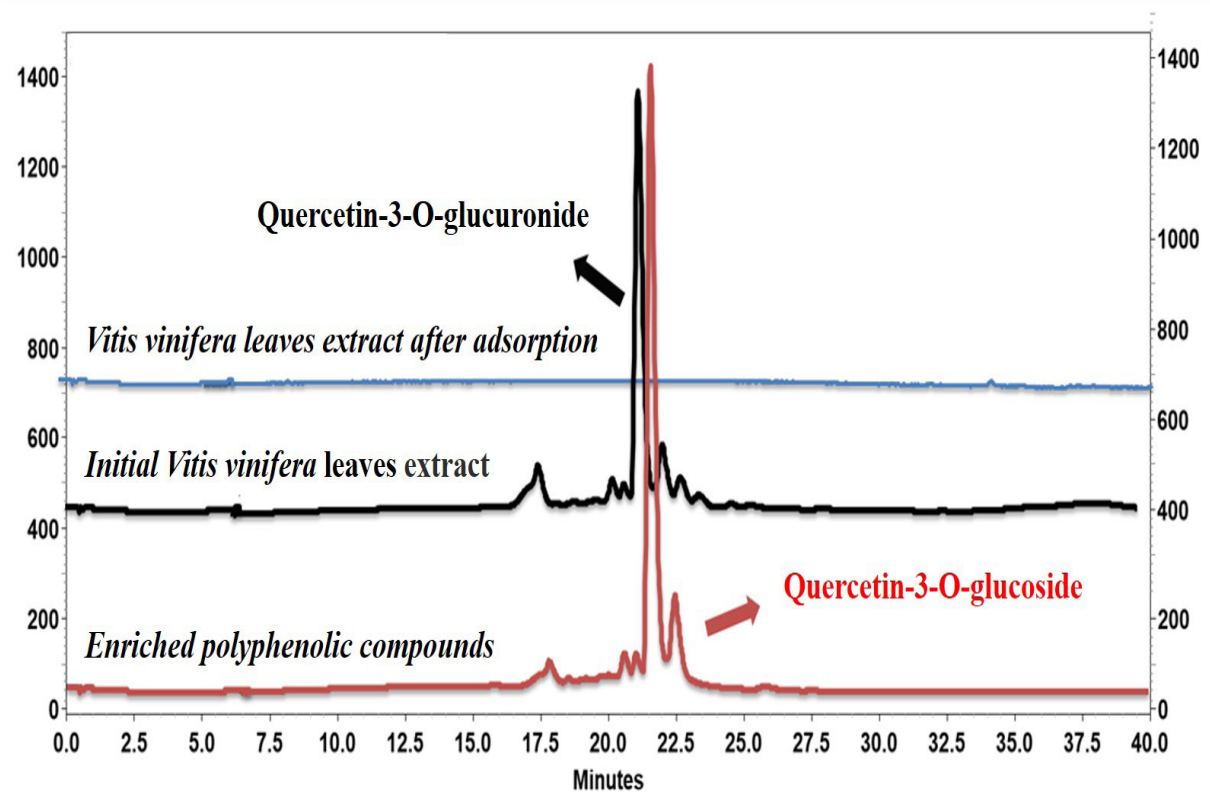

Fig. 5. The HPLC chromatogram of the grape leaves extract before and after adsorption with LXA-10 resin and enriched polyphenolic sample at 355 nm.

antioxidant compounds which can be monitored at 593 nm. Polyphenolic compounds are natural compounds with high antioxidant activity due to a large number of hydroxyl groups in their structures (Bautista-Hernández et al., 2021). The antioxidant activity increased from 252.38 ± 0.04 mM Fe(II).mg^{-1} in initial extract to 408.02 ± 0.02 mM Fe(II).mg^{-1} in enriched polyphenolic compounds of grape leaves extract which shows the 1.61-fold increase in antioxidant activity. Furthermore, the total flavonoid contents of the enriched polyphenolic compounds significantly increase from 227 to 428 mg rutin.g^{-1} sample dry weight which can be effective for increasing antioxidant activity of the enriched powder. This amount is half of the antioxidant activity of ascorbic acid (850 ± 0.02 mM Fe(II).mg^{-1}) and remarkable compared with some polyphenolic extract such as *Peucedanum pastinacifolium* Boiss. & Hausskn aerial part which showed the antioxidant activity 5-fold

lower than ascorbic acid.

4. Concluding remarks

In this project, the adsorption/desorption of grape extract, for the preparation of enriched polyphenolic compounds, was investigated on LXA-10 macroporous resin. For this purpose, the hydroalcoholic extraction was first made using an ultrasound-assisted probe with power 50% for 10 min. In this sense, the adsorption process was performed on LXA-10 resin and the effective parameter in the adsorption process such as pH value was optimized. The pH value of 3.6, as the initial pH of the grape leaves extract, was selected as the optimum pH. Kinetic adsorption process showed pseudo-second-order model with R^2 = 0.99 fitted with adsorption data. In dynamic study, the resin adsorption capacity and required volume for maximum polyphenolic desorption were determined as 42 mL and six-bed volume (BV), respectively. The obtained enriched polyphenolic powder showed an increase of 2.4 and 1.8-fold in the amount of quercetin-3-*O*-glucuronide and quercetin-3-*O*-glucoside, respectively. Moreover, the antioxidant activity of grape leaves extracts also increased from 252.37 to 408.03 mM iron(II).mg^{-1} based on the FRAP test.

Conflict of interest

The authors declare that there is no conflict of interest.

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References

Alam, M.Z., Alhebsi, M.S.R., Ghnimi, S., Kamal-Eldin, A., 2021. Inability of total antioxidant activity assays to accurately assess the phenolic compounds of date palm fruit (*Phoenix dactylifera* L.). NFS J. 22, 32-40.

Bautista-Hernández, I., Aranda-Ledesma, N.E., Rojas, R., Tafolla-Arellano, J.C., Martínez-Ávila, G.C.G., 2021. Antioxidant activity of polyphenolic compounds obtained from *Euphorbia antisyphilitica* by-products. Heliyon 7(4), e06734.

Bayati, M., Manzari Tavakoli, M., Nejad Ebrahimi, S., Aliahmadi, A., Rezadoost, H., 2021. Optimization of effective parameters in cold pasteurization of pomegranate juice by response surface methodology and evaluation of physicochemical characteristics. LWT 147, 111679.

Capello, C., Leandro, G.C., Maduro Campos, C.E., Hotza, D., Mattar Carciofi, B.A., Valencia, G.A., 2019. Adsorption and desorption of eggplant peel anthocyanins on a synthetic layered silicate. J. Food Eng. 262, 162-169.

Chang, T., Bi, Y., Jing, L., Liu, X., Fan, M., Yao, S., Feng, L., Zhao, Y., 2021. Simultaneous adsorption of acid and flavonoids from hawthorn juice onto resins. J. Food Eng. 291, 110195.

Orhan, D.D., Orhan, N., Özçelik, B., Ergun, F. 2009. Biological screening of *Vitis vinifera* L. leaf fractions. Turk. J. Biol. 33(4), 341-348.

Heravi, S., Rahimi, M., Shahriari, M., Ebrahimi, S.N., 2022. Enrichment of phenolic compounds from grape (*Vitis vinifera* L.) pomace extract using a macroporous resin and response surface methodology. Chem. Eng. Res. Des. 183, 382-397 .

Ma, T., Sun, X., Tian, C., Luo, J., Zheng, C., Zhan, J., 2015. Enrichment and purification of polyphenol extract from *Sphallerocarpus gracilis* stems and leaves and *in vitro* evaluation of DNA damage-protective activity and inhibitory effects of alpha-amylase and alpha-glucosidase. Molecules 20(12), 21442-21457.

Meng, X., Gao, N., Zhang, Q., Zhang, X., Li, B., Jiao, X., 2017. Optimation of enrichment and purification of polyphenols from blueberries (*Vaccinium* Spp.) by macroporous resins Xad-7hp. Emir. J. Food Agric. 581-588.

Mohammadi, E., Fattahi, M., Barin, M., Ashrafi-Saeidlou, S., 2022. Arbuscular mycorrhiza and vermicompost alleviate drought stress and enhance yield, total flavonoid concentration, rutin content, and antioxidant activity of buckwheat (*Fagopyrum esculentum* Moench). S. Afr. J. Bot. 148, 588-600.

Moldovan, M.L., Carpa, R., Fizesan, I., Vlase, L., Bogdan, C., Iurian, S.M., Benedec, D., Pop, A., 2020. Phytochemical profile and biological activities of tendrils and leaves extracts from a variety of *Vitis vinifera* L. Antioxidants (Basel) 9(5), 373.

Nielsen, S.S., 2010. Phenol-Sulfuric Acid Method for Total Carbohydrates, in: Nielsen, S.S. (Ed.), Food Analysis Laboratory Manual. Springer US, Boston, MA, pp. 47-53.

Pismenskaya, N., Sarapulova, V., Klevtsova, A., Mikhaylin, S., Bazinet, L., 2020. Adsorption of anthocyanins by cation and anion exchange resins with aromatic and aliphatic polymer matrices. Int. J. Mol. Sci. 21(21), 7874.

Rathi, B.S., Kumar, P.S., 2021. Application of adsorption process for effective removal of emerging contaminants from water and wastewater. Environ. Pollut. 280, 116995.

Seif Zadeh, N., Zeppa, G., 2022. Recovery and concentration of polyphenols from roasted hazelnut skin extract using macroporous resins. Foods 11(13), 1969.

Tacchini, M., Burlini, I., Maresca, I., Grandini, A., Bernardi, T., Guerrini, A., Lerin, L., Sacchetti, G., 2019. Polyphenols from *Vitis vinifera* Lambrusco by-products (leaves from pruning): Extraction parameters evaluation through design of experiment. Nat. Prod. Commun. 14(7), doi: 10.1177/1934578X19862906.

Wieszczycka, K., Filipowiak, K., Aksamitowski, P., Wojciechowska, I., 2020. Task-specific ionic liquid impregnated resin for zinc(II) recovery from chloride solutions. J. Mol. Liq. 299, 112115.